

PATENT

IN THE UNITED STATE PATENT AND TRADEMARK OFFICE

Applicant: Barbizan et al.
S/N 10/561,563
For: Laundry Treatment Compositions
Confirmation No: 9912

Group: 1796
Examiner: B. Mruk

DECLARATION UNDER 37 CFR SECTION 1.132

Hon. Commissioner of Patents
And Trademarks
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Dr. Stephen Norman Batchelor, residing at 37 Hamilton Street, Chester, Cheshire
United Kingdom CH2 3JQ do hereby declare that:

1. I am a citizen of the United Kingdom.
2. My educational and technical background is as follows:
 - a) I received a Bachelor of Arts (Hons) in Chemistry from Oxford University, England in 1990
 - b) I received a Doctor of Philosophy in Physical Chemistry from Oxford University, England in 1993.
 - c) I worked as a Research Fellow at the Free University of Berlin (Germany) Research Fellow 1993 till 1994
 - d) I worked as a Post-doctoral Fellow at Zurich University (Switzerland) from 1994 till 1997
 - e) I have been employed at Unilever since 1997. I am currently a Lead Research Scientist
 - f) I have published 44 academic papers in peer reviewed journals
3. I have read Barbizan et al. U.S. application S/N 10/561,563 filed on 06/08/2006 of which I am the named co-inventor. I have also read the Office Action dated September 8, 2008 and references that was cited: Trimmer, US 3,755,201.

4. The following experiments were carried out under my direction in support of the above-cited patent application. The experiments were done to provide information in response to the most recent Office Action of the present application.

Comparison of Whitening Ability of Bis-Azo Direct Violet Dyes to those of the Prior Art, in Domestic Laundering

Experiment 1

A wash solution was created in demineralised water which contained 1g/L of Linear Alkyl Benzene Sulfonate as a Surfactant (LAS), 1g/L Na_2CO_3 , and 1g/L Na_2SO_4 . The solution was used to wash a piece of bleached, nonmercerized, white, woven Egyptian cotton cloth at a liquor to cloth ratio of 30:1. Washes consisted of agitating the cloth in solution for 30 minutes, then rinsing the cloth twice in water and drying in the air. The experiment was repeated with the addition of dyes to the wash solution. The dyes were added so that the optical density, OD, at the maximum absorption of the dye in the visible range was ~0.1 at 5cm pathlength. The optical density was measured using a UV-visible spectrometer.

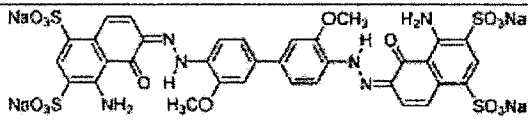
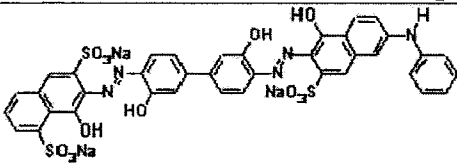
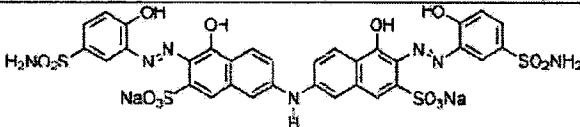
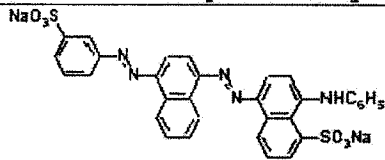
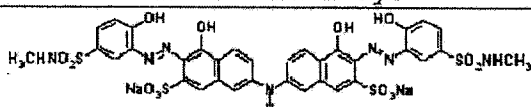
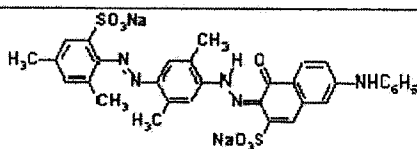
Following the washes the Ganz Whiteness of the clothes were measured. The increase in Ganz whiteness, ΔGanz , due to the dye was calculated according to the equation

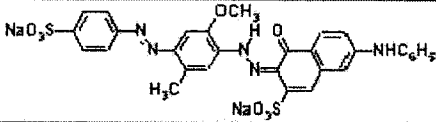
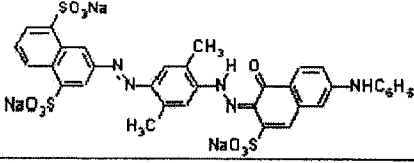
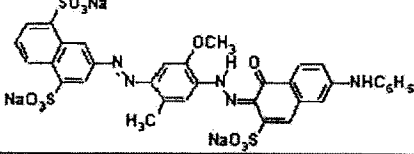
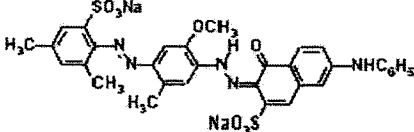
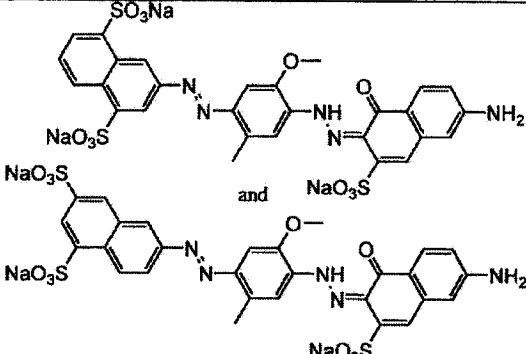
$$\Delta\text{Ganz} = \text{Ganz}(\text{dye}) - \text{Ganz}(\text{control with no dye})$$

In the optical density range 0-0.15, ΔGanz increases approximately linear with optical density.

The dye structures and results are summarized in Table 1 below.

TABLE 1: Comparative Examples
from US3378093, US3755201 (Trimmer), US3762859
(all Colgate)

Dye name	Dye structure	OD	Δ Ganz
Direct Blue 1	 <p align="center">Bis-azo benzidine direct dye</p>	0.11	33
Direct Blue 98	 <p align="center">Bis-azo copper complex direct dye</p>	0.10	26
Direct Violet 66	 <p align="center">Bis-azo Cu complex direct dye</p>	0.12	41
Acid Blue 205	Structure not disclosed acid anthraquinone dye	0.10	2
Acid Blue 113	 <p align="center">Bis-azo acid dye</p>	0.10	14
Direct Violet 48	 <p align="center">Bis-azo copper complex direct dye</p>	0.12	29
Bis Azo Direct Violet dyes of This Invention			
Direct Violet 7		0.12	47

Direct Violet 9		0.12	68
Direct Violet 26		0.11	50
Direct Violet 35		0.10	44
Direct Violet 51		0.12	59
Direct Violet 99		0.11	49

Experiment 2

The optical density (OD) of the dye solutions used for Experiment 1 differed slightly; this is because it is difficult to obtain solutions of exactly the same OD. The delta Ganz increases as the optical density of the dye in solution increases. So for those dyes that seem to give similar performance, one has to compare at the same optical density to make a true comparison of efficacy of the dye as represented by the delta Ganz value. Therefore, Experiment 1 was repeated for the two lowest performing bis-azo dyes, direct violet 7 and direct violet 35 and for the two best performing of the comparative dyes, direct violet 48 and direct violet 66, with the goal to allow comparison of the performance at identical optical densities. In this experiment, the OD was varied between 0 and 0.11 (points at 0, ~0.025, ~0.05, and ~0.1) to obtain a dose response for each dye. The OD was measured for each wash solution before washing.

The dose responses were approximately linear plotting ΔGanz on the y-axis and OD on the x-axis. For each dose response a straight line was fitted, with the intercept through the origin (0,0). The gradient obtained and the R^2 values are given below in Table 2.

TABLE 2

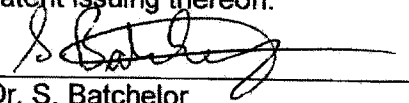
dye	Gradient ($\Delta\text{Ganz}/\text{OD}$)	R^2
Direct violet 7	496	0.97
Direct violet 35	554	0.98
Direct Violet 48 (comparative)	368	0.98
Direct violet 66 (comparative)	442	0.98

R^2 values reflect the goodness of the fit, value close to 1 mean a good correlation.

The greater the gradient indicates the better the whitening performance. The bisazo dyes direct violet 7 and direct violet 35 givesubstantially better whitening performance than the comparative dyes. Direct Violet 7, the poorest performing bis-azo dye of Table 1, was 12% superior to the best performing comparative dye, direct violet 66

5. I conclude from the results in Tables 1 and 2 above that the bis-azo direct violet dyes within the scope of the amended claims result in substantially greater whiteness than the dyes taught by US3378093, US3755201 (Trimmer), US3762859 (Colgate).

6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.


Dr. S. Batchelor

Dated: 15th Jan 2009